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U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

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U.S. APPLICATION NO. (If known, add 37 CFR 1.53)

09/581781

INTERNATIONAL APPLICATION NO.

PCT/US97/24224

INTERNATIONAL FILING DATE

19 December 1997

PRIORITY DATE CLAIMED

TITLE OF INVENTION

Low Environmental Toxicity Latex Coatings

APPLICANT(S) FOR DO/EO/US

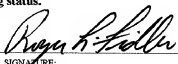
Sugerman, Gerald

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☐ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ has been transmitted by the International Bureau.
 - c. ☒ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☒ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

09/581781 INTERNATIONAL APPLICATION NO	ATTORNEY'S DOCKET NUMBER																				
17. <input type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$970.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$840.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$690.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$670.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) \$96.00 ENTER APPROPRIATE BASIC FEE AMOUNT =	<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="text-align: left;">CALCULATIONS</th> <th style="text-align: left;">PTO USE ONLY</th> </tr> <tr> <td colspan="2" style="height: 100px;"></td> </tr> </table>	CALCULATIONS	PTO USE ONLY																		
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Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	\$																				
<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 20%;">CLAIMS</th> <th style="width: 20%;">NUMBER FILED</th> <th style="width: 20%;">NUMBER EXTRA</th> <th style="width: 20%;">RATE</th> <th style="width: 20%;"></th> </tr> <tr> <td>Total claims</td> <td>7</td> <td>- 20 =</td> <td>X \$18.00</td> <td>\$</td> </tr> <tr> <td>Independent claims</td> <td>3</td> <td>- 3 =</td> <td>X \$78.00</td> <td>\$</td> </tr> <tr> <td colspan="4">MULTIPLE DEPENDENT CLAIM(S) (if applicable)</td> <td>+ \$260.00</td> </tr> </table>	CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		Total claims	7	- 20 =	X \$18.00	\$	Independent claims	3	- 3 =	X \$78.00	\$	MULTIPLE DEPENDENT CLAIM(S) (if applicable)				+ \$260.00	\$ 260.00
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TOTAL OF ABOVE CALCULATIONS =	\$ 930.00																				
Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).	\$ 465.00																				
SUBTOTAL =	\$ 465.00																				
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TOTAL NATIONAL FEE =	\$ 465.00																				
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property	\$																				
TOTAL FEES ENCLOSED =	\$ 465.00																				
	Amount to be refunded: \$ charged: \$																				
a. <input type="checkbox"/> A check in the amount of \$ <u>465.00</u> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. _____. A duplicate copy of this sheet is enclosed.																					
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.																					
SEND ALL CORRESPONDENCE TO:																					
 SIGNATURE: _____ NAME: <u>Roger L. Fidler</u> 28,603 REGISTRATION NUMBER																					

GS 1010

Title:

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. **DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO:** Assistant Commissioner for Patents, Washington, DC 20231.

LOW ENVIRONMENTAL TOXICITY LATEX COATINGSBackground of The Invention:

The use of ammonia and/or volatile amines as neutralizing agents and/or stabilizers, and of alcohols, glycols, and glycol monoethers and monoesters, often in combination, at levels to 40% by volume (exclusive of water) has been employed for more than fifty years to achieve the coalescence of latex solids in acrylic, polyvinyl acetate and related copolymer resins based coatings. The volatilization of these conventional neutralizers, and coalescing components, after achieving film coalescence is normally required in order to inhibit the resultant film's breakdown (reversion) in the presence of humid environments, and to provide acceptable wear and stain resistance to the dried film.

Recent concerns regarding the environmental degradation (predominantly low level ozone formation), and the health and fire hazards associated with exposure to ammonia, volatile amines and volatile organics (VOCs), has led to increasingly strict regulatory limitations on the nature, and proportions, of VOCs which may be employed in coatings. One technique that has been employed in order to comply with said limitations in latex coating applications is the development of self coalescing latex resins which require no coalescents. However, to date, such materials have had the disadvantage of being limited to low Tg film formers with poor performance properties.

Subject of The Invention:

This invention teaches the use of low levels of combinations of nonvolatile reactive amines, in combination with hydroxyl

1 bearing unsaturated esters and/or ethers and/or ether-esters,
2 and/or non-hydroxyl bearing unsaturated esters and/or ethers
3 and/or ether-esters and incompletely etherified and/or esterified
4 oligomeric glycols and/or oligools as partial or full replacements
5 for conventionally employed volatile amines and/or ammonia as
6 neutralizers, and organic solvents as coalescents, respectively,
7 in latex resin applications. This invention has the advantage of
8 reducing emissions and enhancing the performance of films produced
9 from conventional latex resins, and when employed in conjunction
10 with certain types of hypersurfactants (cf. Table 5) also
11 upgrades pigment and/or extender dispersion, and reduces grind
12 times in particulate containing variants; thus enhancing plant and
13 energy use efficiencies. Synergistic performance enhancement, and
14 VOC reduction in latex resins may be attained via the employment
15 of the aforementioned technologies in combination. Partial
16 replacement of either or of both of the aforementioned components
17 by the alternatives of this invention is shown to provide lesser,
18 but still desirable benefits.

19 Preferred Embodiment of The Invention

20 The non-volatile reactive amines useful in the practice of
21 this invention have vapor pressures below 0.1 mm Hg at 25°C,
22 contain at least one basic nitrogen, and at least one carbon to
23 carbon double bond, and/or a transition metal ligand, and contain
24 no more than twelve carbon atoms per basic nitrogen atom. Those
25 more preferable contain one or more (meth)acryl and/or N-vinyl
26 ligands, and those most preferable have a water solubility

1 exceeding 2% at 25°C. Specific examples of such useful non-
2 volatile reactive amines are given in Table (1). These examples
3 are intended to be illustrative rather than exhaustive of the
4 scope of useful materials.

5
6 TABLE I

7	(1A)	N-vinyl pyrrolidone
8	(1B)	N,N,N'-tris (2-butenyl), ethylene diamine
9	(1C)	N', methyl-1, 3-propylene diamine mono 2- propenamide
10	(1D)	N, 2-propenyl, bis (2-hydroxy)propyl amine
11	(1E)	N, 2-propenyl, N'- (2-hydroxy)ethyl, hexamethylene
12		triamine
13	(1F)	4-(N, 3-hydroxypropyl, N-vinyl) 2-amino ethyl 2-butenolate
14	(1G)	2-[N, -(2-oxa-cyclopentadienyl)] amino acetic acid ethyl
15		ester
16	(1H)	4-(N,N bis vinyl) 1,3-pentanediol
17	(1I)	tetraethylene glycol mono 3-(N, ethyl) amino, 2-(methyl)
18		2-propenoate
19	(1J)	N,N-divinyl glutamic acid 2-propenyl ester
20	(1K)	6-(N,N bis vinyl) hexanoic acid ethyl ester
21	(1L)	Titanium IV tetrakis N, 2-aminoethyl ethanolato

22 The preferred types of the hydroxyl bearing, unsaturated
23 esters and/or ethers and/or ether-esters useful in the practice
24 of this invention are those having vapor pressures below 0.1 mm
25 Hg at 25°C, which are capable of air initiated, oxidative
26 oligomerization and/or polymerization derived non-reversible

1 bonding, under normal latex application conditions, to film
2 component(s) and/or to substrate, in order to maximize coating
3 properties via crosslinking the resulting latex thereby
4 minimizing its (post film formation) environmental sensitivity.
5 Examples of such unsaturated esters and ether and ether-esters as
6 are useful in the practice of the instant invention are provided
7 in Table II. These examples are intended to be illustrative
8 rather than exhaustive of the scope of useful materials.

9 Table II

- 10 (2A) trimethylol propane bis (2-methyl)-2-propenoate ester
11 (2B) sorbitan tetrakis 2-butenate ester
12 (2C) bis pentaerythritol 2-propenolato, tris 2-propenoate ester
13 (2D) hexanoic acid 6-hydroxy, (2-propenoato)ethyl ester
14 (2E) citric acid mono isodecenyl ester
15 (2F) malic acid bis cinnamyl ester
16 (2G) 3-heptanoyl furfuryl alcohol
17 (2H) 1,2,3-propanetriol 1-vinyl ether, 2-phenyl carboxylate ester
18 (2J) trimeric 2-butenediol mono (methyl) glutarate ester
19 (2K) ethoxylated (4) bis phenol A mono 2-propenoate ester

20 The preferred types of the non-hydroxyl bearing unsaturated
21 esters and/or ethers and/or ether-esters useful in conjunction with
22 the practice of this invention are those having vapor pressures
23 below 0.1 mm Hg at 25°C, which are capable of air initiated
24 oxidative oligomerization and/or polymerization, and non-reversible
25 bonding, under normal latex application conditions, to film
26 component(s) and/or to substrate, in order to maximize coating

properties via crosslinking the resulting latex thereby minimizing its (post film formation) environmental sensitivity. Examples of such non- hydroxyl bearing unsaturated esters and ether and ether-esters as are useful in the practice of the instant invention are provided in Table III. These examples are intended to be illustrative rather than exhaustive of the scope of useful materials.

Table III

(3A) trimethylol propane bis (2-methyl)-2-propenoate ester, mono vinyl ether

(3B) 1,2,3-propane triol tris 2-butenate ester

(3C) penta erythritol 2-propenolato, tris 2-propenoate ester

(3D) hexanoic acid 6-acetoxy, (2-propenoato)ethyl ester

(3E) fumaric acid bis isodecyl ester

(3F) maleic acid bis cinnamyl ester

(3G) furoic acid vinyl ester

(3H) 1,2,3-hexanetriol 1,2-bis vinyl ether, phenyl carboxylate ester

(3J) trimeric 2-butene-1,4-diol bis propionate ester

(3K) ethoxylated (4) bisphenol A bis 2-(methyl)-2-propenoate ester

The preferred types of the incompletely etherified and/or esterified oligomeric glycols and/or oligools useful in the practice of this invention are those having vapor pressures below 0.1 mm Hg at 25°C, which are oligomers of two to 4 carbon diols, and of three to six carbon triols, wherein each of the ether and/or ester ligands contains five or fewer carbon atoms per oxygen.

1 Examples of such incompletely etherified and/or esterified
2 oligomeric glycols and/or oligools as are useful in the practice of
3 the instant invention are provided in Table IV. These examples are
4 intended to be illustrative rather than exhaustive of the scope of
5 useful materials.

6 Table IV

- 7 (4A) penta (ethylene glycol) mono methyl ether
8 (4B) tetra (1,4-butylene glycol) mono (2-methyl) butyrate
9 (4C) 1-hydroxy-2,5-bis methyl-3,6,9,12-tetraoxa tetradecane
10 (4D) 4-oxaheptane-1,2,6,7-tetraol mono acetate, mono 2-propyl
11 ether (mixed isomers)
12 (4E) ethoxylated (6) 1,2,4-butanetriol bispropanoate (mixed
13 isomers)
14 (4F) tris 1,2,5-n pentane triol tetraethyl ether (mixed isomers)
15 (4G) tris (1,3-propane-diol) mono isopentyl ether
16 (4H) 1,2- bis (2- hydroxy ethoxy ethyl) 1,2,3-propane triol
17 (4J) polyethylene glycol (300) mono amyl ether
18 (4K) tris neopentyl glycol mono n-propyl ether

19 The surfactants most useful in the practice of this invention
20 are those having vapor pressures below 0.1 mm Hg at 25°C, which are
21 capable of non-reversible bonding, under normal processing
22 conditions, to film component(s) and/or substrate in order to
23 maximize coating properties, while minimizing post film formation
24 environmental sensitivity, and which serve to efficiently wet
25 substrates coated, and to disperse particulates, if any, employed
26 in the formulated latex coating. Among the surfactants found to be

1 useful in the practice of this invention are amphoteric detergents,
2 and certain organometalics based on tetravalent titanium or
3 zirconium. These last have been found to contribute significantly
4 to substrate adhesion and improved corrosion resistance on wood,
5 metallic and ceramic substrates, and to be particularly useful in
6 maximizing color intensities of carbon black, azo and
7 phthalocyanine based pigments. Specific examples of the preferred
8 types of hypersurfactants are given in Table V. These examples are
9 intended to be illustrative rather than exhaustive of the scope of
10 useful materials.

11 Table V

- 12 (5A) 12-N,N,N-trimethylaminododecanoato
13 (5B) N-(pentakis oxyethylene sulfato) triethylene diamine
14 (5C) p-[6-N(methyl) morpholino]octyl phenyl phosphonic acid
15 (5D) N,N,N-triethyl glutamic acid
16 (5E) titanium 4 octyl, [(tris octyl) diphosphato
17 (5F) titanium 4 oxoethylene, bis (dodecyl) phenylsulfonato
18 (5G) oxy [bis titanium 4 (bis tridecyl) diphosphate]
19 (5H) zirconium 4 tetraethylene glycol monomethyl ether, tris
20 (tetraethylene glycol monomethyl ether) diphosphato
21 (5J) zirconium 4, bis w-N,N-(dimethyl)amino octanoato, 1,4-
22 cyclohexanediolato
23 (5K) triethylene glycol diolato, bis [zirconium 4 tris (octyl)
24 phosphate]

25 Those skilled in the art shall no doubt be capable of
26 subverting the teachings of this invention via the substitution of

1 functionally equivalent materials, e.g. employment in combination
2 of hydroxylated and non-hydroxyl bearing esters (ether-esters)
3 optionally in combination with partially esterified and/or
4 etherified glycols, and or polyols, in place of either component
5 (set) alone, (as is amply demonstrated in example No. 4.), or of
6 unsaturated analogous unsaturated amides, for a portion of the
7 aforementioned unsaturated ethers, esters or ether-esters disclosed
8 above, and/or fluorination of one or more of the species of
9 components heretofore described as necessary to the successful
10 practice of this invention; however such non-critical
11 modifications, and/or combinations of relevant species types, must
12 be considered as within the scope of this disclosure.

13 Further amplification of the scope and utility of the instant
14 invention to latex coating applications in inks, paints and stains
15 shall be found to be illustrated by the content(s) of examples 1
16 through 5. Said examples are intended to be illustrative rather
17 than exhaustive of the extraordinarily diverse applicability of the
18 instant invention.

19 Example #1

20 This example teaches the superiority of the present invention
21 versus the prior art with respect to the productivity, VOC
22 emissions, and performance quality in a masonry sealer application.

23 A masonry sealer formulation was prepared by the sequential
24 dispersion of the indicated components (pigment dispersion times
25 and grind quality achievement was noted). The resulting sealer was
26 applied via roller to smooth surface, ten day old, 8" X 18" X 1"

thick concrete castings, at an application rate of one gallon per 400 square foot, dry time (to touch) was measured under conditions of 72° F and 85% humidity. After 240 hours of drying @ 72° F and 85% humidity, sealer performance was measured by weighing the dry casting, then impounding a 6" depth of water, or alternatively 6% salt solution, on such a casting for twenty four hours, then draining and weighing the drained casting. The weight percent of water, and independently that of 6% salt solution, adsorbed by said castings were used to determine sealer efficacy. The results of this study are given in Table No. 1.

Formulation: in parts by weight; (in order of addition) water 200.0; neutralizer¹, as shown; surfactant^a, as shown, biocides², 18.50; hydroxy ethyl cellulose, 5.00; potassium tris polyphosphate, 2.00; defoamer², 1.00; coalescent^a, as shown; ultramarine blue pigment, 0.25; rutile titanium dioxide, 200.0, American process zinc oxide, 25; platey talc, 250; water, 49.98; AC-625 Acrylic latex resin³, 352.0; defoamer², 0.98; surfactant^b, coalescent^b, as shown,; water, 24.99; and sodium nitrite 2.30; thixotrope⁴, as shown (required) to adjust system viscosity to 85-90 KU at 75° F.

Table No. 1

<u>Formulation</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
neutralizer	amp-95/1.98	1B/2.00	1H/1.80	1G/2.20	1D/1.78
surfactant(a)	Tamol 850 ⁷ /14.85	5A/1.80	5E/1.75	5K/1.55	5H/1.50
coalescent(a)	Propylene glycol /34.56	none	none	4J/4.50	none
surfactant(b)	Triton N101 ³ /2.20	none	5B/1.00	none	5D/1.25
coalescent(b)	Texanol ⁷ /9.88	2A/16.42	3A/12.60	3F/18.55	2H/11.70

<u>Formulation</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>
neutralizer	ammonia/1.00 1L/1.00	1B/1.00 ammonia/1.00	1H/1.80 AMP-95/1.00	1G/1.20	1D/1.78
surfactant (a)	Tamol 850 ⁷ /14.85	5A/1.80	5E/0.75	5K/1.55	5H/0.75
surfactant (b)	Triton N101 ⁷ /2.20	none	Triton N101 /2.20	none	5D/1.25
coalescent (a)	Propylene glycol /34.56	none	none	4J/4.50	Propylene glyco /17.88
coalescent (b)	Texanol ⁸ 19.98	2A/16.42	3A/12.60	3F/18.55	2H/5.85

<u>Formulation</u>	<u>VOC g/l⁵</u>	<u>Dispersion time hr.</u>	<u>Grind Hegman</u>	<u>Water adsorption g.</u>	<u>Salt water adsorption g.</u>
1	124	3.4	4	46	61
2	8	0.7	6	27	32
3	3	0.6	6	21	28
4	5	0.7	5	24	27
5	4	0.6	6	25	30
6	112	3.1	4	37	48
7	10	0.7	6	32	42
8	3	2.4	5	27	38
9	6	0.7	5	24	27
10	36	1.8	6	31	39

Notes: a) As shown; 1) A combination of 3.5 parts of Nuosept 95, and 15 parts of Nuocide 404D, Huls Corp. were employed; 2) Defo 806-102; Ultra Inc. 3) AC-625, Union Carbide Corp.; 4) Rhevis CR, Rhevis Corp. 5) via EPA Method 24GC; 6) Rohm and Haas Corp.; 7) Eastman Kodak Inc.

The efficacy of the coalescent systems of the instant art in producing a less water and salt permeable, acrylic latex based masonry seal coating, is compared to a conventionally coalesced counterpart, Formulation No. 1, and is obvious from the preceding data. VOC emissions reduction and improvement in both productivity and dispersion level achieved are likewise self evident.

Example #2

This example teaches the superiority of the present invention versus the prior art with respect to the productivity, VOC

1 emissions, and performance quality in a direct to metal,
2 maintenance coating application.

3 Direct to metal coatings were prepared by the sequential
4 dispersion of the indicated components (pigment dispersion times
5 were noted). The resulting coating was spray applied to sandblasted
6 smooth surface 24" X 8" carbon steel test panels at application
7 rate of one gallon per 250 square feet. After 120 hours of drying
8 @ 72° F and 85% humidity, edge sealing and scribing, the coatings'
9 corrosion resistance performance were each measured by QUV cabinet
10 exposure [cyclic exposure to UV radiation, 4% saline solution, and
11 varying temperature (25°-80° C)].

12 **Formulation:** in parts by weight (in order of addition): water,
13 50.0; neutralizer, as shown; Surfactant^a, as shown, biocide¹, 4.00;
14 oxidized polyethylene wax, 4.00; (disperse wax) polyurethane
15 thixotrope², as shown; defoamer³, 2.00; coalescent^a, as shown,
16 ultramarine blue pigment, 0.25; rutile titanium dioxide, 125; zinc
17 aluminate 150; Acrylic latex resin⁴, 64.0; (disperse particulates
18 to Hegman 7.5+). Neutralizer, is shown; acrylic latex resin⁴,
19 564.0; defoamer², 0.98; surfactant^b, coalescent^b, as shown; water,
20 16.00; arid sodium nitrate 2.30. Thixotrope², (as required) to
21 adjust system viscosity to 80-85 KU at 75° F . The control coating
22 required 3.7 hours to disperse to a Hegman grind gauge reading of
23 7+, whereas each of the instant art coatings achieved said fineness
24 of grind in less than one half hour. The results of this study are
25 given in **Table No. 2**

Table No. 2

<u>Formulation</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
neutralizer	DMAMP-80 ⁵ /12.5	1A/4.00	1C/1.80	1E/2.20	1J/1.78	DMAMP-80 ⁵ /6.25 1A/ 2.00
surfactant (a)	Triton CF10 ⁶ /10.00	5D/1.80	5F/1.75	5C/1.55	5J/1.50	Triton CF 10/5.00
thixotrope ²	15.00	5.00	5.50	8.70	5.30	9.32
coalescent (a)	Dipropylene glycol /34.60	4B/12.00	4E/10.5	4K/8.25	none	none
surfactant (b)	none	none	5A/1.00	Triton CF10 ⁶ /4.0	none	5D/1.25
coalescent (b)	PmPE ⁷ /44.60	2A/16.42 4A/7.45	3A/12.60	3F/18.55	2H/14.70	2H/14.70
thixotrope ²	21.40	3.20	1.50	0.70	2.30	1.95
<u>Formulation</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
neutralizer	DMAMP-80 ⁵ /12.5	1A/4.00	1C/1.80	1E/2.20	1J/1.78	DMAMP-80 ⁵ /6.25
surfactant (a)	Triton CF10 ⁶ /10.00	5D/1.80	5F/1.75	5C/0.55	5J/1.50	Triton CF10/5.00
surfactant (b)	none	none	5A/1.00	Triton CF10 ⁶ /2.0	none	5D/1.25
thixotrope ²	15.00	5.00	5.50	8.70	5.30	9.32
coalescent (a)	Dipropylene glycol /17.3	PmPE ⁷ /22.60	4B/12.00	4E/10.5	4K/8.25	PmPE ⁷ /22.3
coalescent (b)	2A/ 8.81	2A/ 6.42	PmPE ⁷	3F/ 18.55 4A/7.45	2H/14.70	2H/14.70
thixotrope ²	18.90	6.20	4.50	3.70	2.30	6.95

<u>Formulation</u>	<u>VOC g/l⁸</u>	<u>Initial gloss</u>	<u>60° gloss @</u>	<u>60° gloss @</u>	<u>60° gloss @</u>
		<u>@60°</u>	<u>200hr. QUV</u>	<u>500 hr. QUV.</u>	<u>1,000 hr. QUV</u>
1	232	82	76	31	film destroyed
2	9	91	87	82	76
3	11	93	90	88	80
4	8	87	86	82	61
5	10	88	86	85	84
6	13	84	80	66	59
7	72	85	79	46	12
8	57	80	76	51	18
9	28	87	84	72	63
10	7	88	86	84	78
11	8	92	90	87	85
12	77	85	80	75	48

Notes: 1) Nuosept 95, -Huls Corp. 2) Acrysol RM 2020, Rohm and Haas 3) Defo 3000; Ultra Inc. 4) HG 56, Rohm and Haas Corp. 5) 80%

1 2-N,N-dimethylamino-2-methyl propanol aq. 6) Union Carbide Corp. 7)
2 propylene glycol mono phenyl ether. 8) via EPA Method 24GC

3 The efficacy of the coalescent systems of the instant art in
4 producing a more environmentally resistant, acrylic latex based
5 direct to metal coating, as compared to a conventionally coalesced
6 counterpart, Formulation No. 1, is obvious from the preceding data.
7 VOC emissions reduction, and improvement in productivity achieved
8 are likewise self evident.

9 Example #3

10 This example teaches the superiority of the present invention
11 versus the prior art with respect to productivity, VOC emissions,
12 and performance quality in a polyvinyl acetate based interior flat
13 architectural paint application.

14 Interior flat paints, were prepared by the sequential
15 dispersion of the indicated components (pigment dispersion times,
16 and dispersion efficacy were noted). The resulting coating was
17 brush applied to unprimed drywall (gypsum sheet) @ 72° F and 80%
18 humidity, coverage, stain removal, and scrubability performance
19 were each measured after 7 days of drying 72+/- 2°F @ 65-80%
20 humidity.

21 Formulation; in parts by weight; (in order of addition) water,
22 200.0; neutralizer¹, as shown; surfactant^a, as shown, biocides²,
23 1.00; hydroxy ethyl cellulose, as shown; potassium tris
24 polyphosphate, as shown; defoamer³, 1.00; coalescent^a, as shown;
25 ultramarine blue pigment, 0.25; rutile titanium dioxide, 250.0,
26 water washed clay⁴, 50.0; calcium carbonate⁵, as shown; diatomite⁶,

1 50.0; water, 49.98; PVA latex resin⁷, 352.0; defoamer², 0.98;
 2 coalescent^b, as shown ; water, 100 .0; and sodium nitrite 2.30;
 3 thixotrope⁶, as shown (required) to adjust system viscosity to 90-
 4 100 KU at 75° F.
 5 The results of this evaluation are shown in Table No. 3.

Table No. 3

Formulation	1	2	3	4	5	6
neutralizer	28% ammonia aq. 6.05	1B/2.00	1H/1.80	1G/2.20	1D/1.78	28% ammonia 6.05
HEC (QP-4400)	5.50	1.20	1.35	1.25	1.40	1.35
surfactant (a)	Tamol 731/6.90 Triton N101/3.31	5E/1.80	5J/1.75	5C/1.55	5F/1.5	Tamol 731/6 Triton N101/3.31
coalescent (a)	Propylene glycol Texanol/9.88 50 /51.95	4A/6.00	4 F/7.00	4H/5.50	4E/7.00	4E/7.00
Calcite	50	150	125	150	140	50
coalescent (b)	none	2A/26.4	3C/12.60	3F/18.50	2J/11.90	2J/11.90
thixotrope ⁶	3.5	3.0	3.1	2.7	2.4	3.9
Formulation	7	8	9	10		
neutralizer	28% ammonia aq. 6.05	1B/2.00	1H/1.80	1H/1.80		
HEC (QP-4400)	5.50	1.20	1.35	1.25		
surfactant (a)	Tamol 731/ 6.9 Triton N101/ 3.31	Tamol 731/ 3.9 5J/1.0	5J/ 1.75	Tamol 731/ 6.9 Triton N101/ 3.		
coalescent (a)	Propylene glycol 51.95	4A/6.00	Propylene glycol 51.95	4H/5.50		
calcite	50	100	125	150		
coalescent (b)	4E/2.00	2A/ 26.4	3C/ 12.60	3F/ 18.50		
thixotrope ⁶	3.5	3.0	3.1	2.7		

Formulation	VOC g/l ³ hr.	Dispersion Time hr.	Grind Hegman	Min.Coalescence temp °C	Scrubs ¹⁰	Stain Removal ¹¹
1	199	2.4	4	47	410	6
2	8	0.4	5	34	1,740	9
3	3	0.6	6	32	2,025	10
4	5	0.6	6	36	1,960	9
5	4	0.5	5	30	2,230	10
6	15	2.2	4	31	785	8
7	190	2.4	4	43	850	7
8	9	0.9	5	35	1,140	8
9	188	1.1	5	45	890	7
10	8	2.4	4	37	1,310	8

Notes: 1) As shown; 2) Nuosept 95, Huls Corp.; 3) Defo 3000; Ultra
 Inc.. 4) 70C Huber Corp. 5) Camel Carb., Cambel Corp.; 6) Diafil 530

viscosity of 65-70 KU, and the coating was applied by curtain coating on sanded but unprimed 4' X 8' X 0.25" laminate natural oak (on pine) substrate. The coated panels were force dried by passing same through a 180° F oven for 20 minutes, then cooled at ambient temperature (ca. 80° F) for 24 hours prior to evaluation, for abrasion and solvent resistance. to determine coating performance, efficacy. The results of this study are given in Table No. 4.

Table No. 4

<u>Formulation</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>
<u>Part A:</u>								
neutralizer	28%	1A	1H	1H	1H	1B	1B	1B
	ammonia aq.							
<u>Part B: formulation</u>								
Texanol	8.00						4.00	4.00
diethylene glycol	18.20						9.10	9.10
mono butyl ether								
2A		12.05	6.00		6.00	6.00	6.00	
2C			4.40	10.70				
3B					4.20			6.00
4G			1.90	2.90	1.05	6.20		
4J								
<u>Formulation</u>	<u>VOC g/l¹</u>	<u>abrasion</u>	<u>solvent</u>	<u>stain</u>	<u>Mix pot life</u>			
	<u>resistance²</u>	<u>resistance³</u>	<u>resistance⁴</u>	<u>hr. @ 80°F⁵</u>				
1	165	114	lifts	poor	7.5			
2	3	31	slight softening	good	42			
3	4	29	slight softening	fair	35			
4	3	24	no change	excellent	40			
5	3	26	no change	good	61			
6	4	98	mod. Softening	fair	46			
7	83	19	slight softening	fair	21			
8	85	78	severe softening	poor	11			

Notes: 1) By EPA Method 24GC; (formulations 2 through 7 produced 0 to negative VOC readings by EPA Methods 24, and 24A; 2) Tabor CS-10 wheel 1000 cycles; 3) 24 hr methyl ethyl ketone covered spot test; 4) 24 hr exposure to lipstick; 5) time to 10% loss of abrasion resistance in finished coating.

This example demonstrates, that the use of the combination of components cited as the basis of the instant invention, viz. That

1 Whittaker, Clark, and Daniels Inc. 7) Rhoplex 3077, Rohm and Haas
2 Corp.; 8) Rhevis CR, Rhevis Corp. 9) via EPA Method 24GC. 10) ASTM
3 method; 11) ASTM method.

4 The efficacy of the coalescent systems of the instant art in
5 producing a more, scrub and stain resistant PVA latex based
6 interior flat architectural coating as compared to a conventionally
7 coalesced counterpart, Formulation No. 1, is obvious from the
8 preceding data. VOC emissions reduction and improvement in both
9 productivity and dispersion level achieved are likewise self
10 evident, as is a considerable reduction in minimum coalescence
11 temperature, without recourse to the use of low boiling, flammable
12 solvent(s), normally employed to induce same.

13 Example #4

14 This example teaches the superiority of the present invention
15 versus the prior art with respect to the productivity, VOC
16 emissions, and performance quality in a force dried, clear,
17 protective, two component acrylic latex cured - waterborne epoxy,
18 wood cabinet coating.

19 Component A. neutralizing agent, as shown-3.5 PBW; sodium
20 nitrite 0.15 and defoamer (Patcote 519-Patco Coatings Inc.) were
21 admixed with 95.85 PBW of (Acrylic latex-Maincote AE 58), and said
22 emulsion was subsequently mixed with 50 PBW of Component B,
23 formulated by blending various additives, as shown, into 12.5 PBW
24 of Genepoxy 370-H55- Daubert Chemical Co., and diluting as
25 necessary with water to produce a total part B weight of 25 parts.
26 Thixatrope¹, was added, as required, to provide an initial mix

1 nonvolatile reactive amines, in combination with hydroxyl bearing
2 unsaturated esters and/or ethers and/or ether-esters, and/or non-
3 hydroxyl bearing unsaturated esters and/or ethers and/or ether-
4 esters and incompletely etherified and/or esterified oligomeric
5 glycols and/or oligools as (partial or full) replacements for
6 conventionally employed volatile amines and/or ammonia as
7 neutralizers, and organic solvents as coalescents, respectively may
8 be employed to substantially enhance the processability (pot life),
9 mechanical and chemical resistance properties (abrasion and stain
10 resistance respectfully) as well as attain VOC reduction in wood
11 coatings. A further benefit of the instant invention as applied to
12 wood coatings is that unlike conventionally coalesced waterborne
13 coatings, e.g. formulation D-1 latex formulations based upon the
14 teachings of this invention, e.g. formulations D-2 through D-7, do
15 not cause significant grain rise, thereby virtually eliminating the
16 necessity for intercoat sanding. These data also demonstrate that
17 subsets of the preferred combination of components herein disclosed
18 (e.g. formulations D-3, D-5, and D-6) may provide considerable
19 benefits relative to their conventional counterparts, such as
20 Formulation No. 1; however, omission of one or more of the
21 components of the combination herein disclosed leads to inferior
22 results as compared to the inclusion of the full compliment.

23 Example #5

24 This example teaches the superiority of the present invention
25 versus the prior art with respect to the productivity, VOC
26 emissions, and performance quality in waterborne flexographic inks.

A latex flexographic ink formulation, was prepared by the sequential dispersion of the indicated components (pigment dispersion times and grind quality achievement was noted). The resulting ink was applied via a #6 wire wound rod to bond paper, and permitted to dry. Dry time (to touch) was measured under conditions, of 72° F and 85% humidity. After 6 hours of drying @, 72° F and 85% humidity, heat seal resistance performance (face to face) was measured at 25 psig. and 2 seconds contact time) the results of this study are given in Table No. 5.

Formulation: in parts by weight; (in order of addition) E-2350 resin 267; neutralizer, as shown; surfactant, as shown; Defo 1020 defoamer 4.00 Ultra Inc.; coalescent^a, as shown; calcium lithol pigment 50% presscake, 400-Sun Chemical Corp.; Michemlube wax 5-Michelman Inc., water, as required in order to produce a viscosity of 27 seconds using a #2 Zahn cup. Relative dry ink color intensities were measured by integrating thin film reflectance spectra at 300-600 um wavelengths using a spectrophotometer after 48 hours of drying at the above conditions

Table No. 5

<u>Formulation</u>	<u>1</u>	<u>2</u>	<u>3</u>
neutralizer	28% ammonia aq, 2.00	1D/2.00	1H/1.40
surfactant	Tamol 850/4.95	5A/1.80	5E/1.75
coalescent	isopropanol/48 Texanol/24	2A/10.5 3C/15.5 4H/5.5	2E/20.0
<u>Formulation</u>	<u>4</u>	<u>5</u>	<u>6</u>
neutralizer	1F/2.00	1D/1.46	28% ammonia aq/2.00
surfactant	5K/1.55	5H/1.50	Tamol 850/ 4.95
coalescent	3C/25.0	3C/18.0 4H/7.0	3C/18.0 4H/7.0

<u>Formulation</u>	<u>VOC g/l⁵</u>	<u>Dry to touch time</u> <u>hr.</u>	<u>6 hr. Heat seal</u> <u>resistance °C</u>	<u>Relative color</u> <u>intensity.</u>
1	144	3.4	86	1.00
2	4	0.7	154	1.42
3	3	0.6	172	1.27
4	5	0.7	104	1.08
5	4	0.6	167	1.39
6	7	0.6	122	1.03

Notes: The efficacy of the coalescent systems of the instant art in producing a faster drying, more strongly colored, and lower VOC acrylic latex based printing ink coating as compared to a conventionally coalesced counterpart are obvious from the preceding data, as are indications that incomplete application of the teachings of this disclosure may lead to inferior results. Note the deficiencies in the heat seal performance of formulation E-4 as compared to E-2, E-3, and E-5, the benefits of hyperdispersant use as indicated E-2 to E-5 vs. E-6.

Example #6

This example teaches the utility of the instant invention in the production of superior waterborne anti-scuff overprint coatings for graphic arts applications. A 30% solution of water reducible styrene-acrylic copolymer resin in water was prepared by admixture of the indicated neutralizing agent-as shown, water, and Air Product Corp's Flexbond 28 resin. Thirty parts by weight (PBW) of the preceding solution, were admixed with 50 PBW of styrene- acrylic latex resin (Flexbond 285, Air Products Corp.), coalescent, and surfactant(s) -as shown, poly ethylene wax, 2 PBW, and sufficient water to dilute the system to 100 PBW.

The resulting coating was applied, in line on a high speed six color lithographic cold web press, to a solid four color print pattern, followed by in line infrared drying, and folding.

Measurements of VOC (by EPA Method 24), offset, and blocking limited maximum allowable impression rates (impressions/ minute [IPM]), were made. The results are given in Table 6.

Table 6

<u>Formulation</u>	<u>1</u>	<u>2</u>	<u>3</u>
neutralizer	28% ammonia/ 6.00	AMP 95 ¹ / 8.00	AMP 95/ 8.00
coalescent (s)	isopropanol/ 12	isopropanol/ 12	2G/ 4.50
surfactant	Tamol 850/ 2.05	Tamol 850/ 2.05	5K/ 0.75
<u>Formulation</u>	<u>4</u>	<u>5</u>	<u>6</u>
neutralizer	1G/ 6.00	1G/ 6.00	1G/ 6.00
coalescent (s)	2G/ 4.50	2G/ 4.50	3C/ 3.65
surfactant	Tamol 850/ 2.05	5K/ 0.75	4C/ 0.85
			2K/ 0.75
<u>Performance</u>	<u>1</u>	<u>2</u>	<u>3</u>
VOC -g/l.	131	146	27
max IPM (offset)	8,240	9,235	12,720
max IPM (block)	10,150	9,950	14,610
<u>Performance</u>	<u>4</u>	<u>5</u>	<u>6</u>
VOC -g/l.	3	4	4
max IPM (offset)	16,670	18,000 ²	18,000
max IPM (block)	17,130	18,000	18,000


1 Claims:

2 What we claim is:

- 3 1) Compositions of matter comprising:
4 essentially nonvolatile reactive amines;
5 hydroxyl bearing, unsaturated esters and/or ethers and/or
6 ether-esters; and/or
7 combinations of non-hydroxyl bearing, unsaturated esters
8 and/or ethers and/or ether-esters, and/or saturated hydroxyl
9 bearing etherified and/or esterified oligomeric glycols
10 and/or oligools.
- 11 2) Compositions of matter comprising:
12 volatile amines and/or ammonia neutralizers;
13 hydroxyl bearing, unsaturated esters and/or ethers and/or
14 ether-esters; and/or
15 combinations of non-hydroxyl bearing, unsaturated esters
16 and/or ethers and/or ether-esters, and/or saturated hydroxyl
17 bearing etherified and/or esterified oligomeric glycols
18 and/or oligools.
- 19 3) Compositions of matter comprising:
20 essentially nonvolatile reactive amines; and
21 organic solvent coalescents.
- 22 4) Compositions of matter as defined in Claims 1, 2 and 3 having
23 as an additional component latex resin.
- 24 5) Compositions of matter as defined in Claims 1, 2, 3 and 4
25 having hypersurfactants as an additional component.
- 26 6) Compositions of matter as defined in Claim 5 in which the

hypersurfactants are derived from titanium or zirconium based organometallics.

- 7) Compositions of matter as in Claim 4 in which the latex resin is derived from poly vinyl acetate and/or acrylic and/or a copolymer thereof.

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DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION (37 CFR 1.63)	Attorney Docket Number <i>GS 1010</i>
	First Named Inventor <i>Sugerman, Gerald</i>
	COMPLETE IF KNOWN
	Application Number <i>PCT/US97/24224</i>
	Filing Date <i>19 December 1997</i>
<input checked="" type="checkbox"/> Declaration Submitted with Initial Filing	<input type="checkbox"/> Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required)
Group Art Unit	
Examiner Name	

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Low Environmental Toxicity Latex Coatings

the specification of which (Title of the Invention)

☐ is attached hereto
OR

☒ was filed on (MM/DD/YYYY) *12/19/2000* as United States Application Number or PCT International

Application Number: *PCT/US97/24224* and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

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			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
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(Page 1 of 2)

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U.S. Parent Application or PCT Parent
Number

9CT/US97/24224

Parent Filing Date
(MM/DD/YYYY)

12/19/97

Parent Patent Number
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☐ A petition has been filed for this unsigned inventor

Given Name (first and middle (if any))

Gerald

Family Name or Surname

Sugerman

Inventor's
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Gerald Sugerman

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